Optimization of pectin extraction from kepok banana peels (*musa paradisiaca*) using surface response methodology

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Abstarct. The experimental design in this study was used to optimize the results of extracting pectin from kepok banana peels by surface method. The model for predicting and optimizing the process of extracting kepok banana pectin consists of 4 axillary points, 4 factorial points, and 5 middle titlication replications using a central commision design (CCD) with DX6.0.4 software. The independent variables used in the optimization of the extraction results of kepok banana pectin are pH and temperature, where the pH is between 1.5 - 2.5 and temperatures are 60°C-100°C and the combination of independent and bound variables (extraction time). The results of pectin extract ranged from 5.79% to 22.57% (b / b, based on the dry weight of kepok banana peel). The variables of pH and temperature, the interaction between pH and temperature, and the interaction between extraction time and pH significantly influence. The optimum conditions for extraction of pectin are estimated at pH (2.5) and temperature (100°C). In optimal conditions, the actual pectin yield is 22.57%, which is below the estimated extraction condition of 21.286%. Analysis of variance, adj R^2 and R^2, model lack of fit test, and p value statistically indicate that the model is adequate in representing experimental data. The effect of pH and extraction temperature was very significant ($P < 0.001$) on the extraction results of kepok banana peel pectin. This shows that these two variables are very important in the process of extracting kepok banana peel, where the R^2 value is 96.01%.

1. Introduction

The main structural components of the primary cell wall and middle lamella of high-level plants that are found in heteropolysaccharides are known as Pectin [1]. This structure contributes to structural strength and plant tissue defence and various cell functions, defense and cell adhesion [2]. Pectin is primarily made up of D-galacturonic acid joined by α - (1- 4) glycosidic linkages [3]. According to [4] pectin must contain at least 65% galacturonic acid. One characteristic of the degree of esterification (DE) is that it affects the application of pectin because it determines the pectin-forming properties. Classification of high methyl esters (HM) pectin shows that the percentage of DE is above 50% while low methyl ester (LM) pectin is less than 50%.

Pectin plays a very important role as a stabilizer and thickener in the production of foods and beverages such as jam, jelly, yogurt, fruit juice, and candy products [5]. According to [6], pectin has been recommended as a complementary food (GRAS) by a joint FAO / WHO committee and its use is

permitted throughout the world. Based on the actual arrangement for each type of plant, the extraction method used will vary [3]. According to [7], the yield and quality of pectin depends largely on the source of the material such as fruit and vegetable waste and the method used for extracting pectin in various mineral acids or saline solutions at certain pH and temperature.

According to [8], bananas (*Musaceae*) are one of the Moses herbaceous plants of the genus. Based on some researchers, it is stated that banana tree cultivation is the fourth largest food crop in the world that grows in every tropical and subtropical region, where the fruit is used in various food industries. About 85% of commercial pectin in the world is used to make jelly, nutrition, cosmetics and pharmaceuticals, gels and emulsifiers for jam, soft drinks, fish and dairy products [9;10].

In the present study, the kepok banana peel waste which was utilized as the source of pectin. It aimed to optimize method in terms of extraction of pH and temperature by the Response Surface Methodology (RSM) with Central Composite Design (CCD) in this case pH and temperature which is the result of the screening significance of the two levels factorial experimental.

2. Method

2.1. Preparation of Materials for Banana peel Extract

Fresh banana peel is collected from the Pa'baeng-baeng market, Tamalate Sub-district, Makassar City, South Sulawesi. Fresh ingredients from kepok banana peel. Banana peel selected from ripe bananas in yellowish green. Furthermore, the peel that has been isolated from the banana bark section was used as the experimental material in this study. Kepok banana peel is cut into small size and washed, then dried in an oven at temperature of 60°C for 5 days until the dry weight of kepok banana peel is stable with a humidity of 7.8%. These dried banana peel samples were milled and filtered to obtain small particle sizes using a 1 mm filter and stored at room temperature in a plastic bag before being used for further research.

2.2. Pectin Extraction

A total of 10 g of kepok banana peel powder was measured by scales and mixed with 400 ml of distilled water (1:40) and acidified with 0.5 N hydrochloric acid to meet the designed pH (1.5; 2.0; 2.5). The mixture is then stirred using a stirrer until evenly distributed, then incubated at a temperature (60 ° C; 80 ° C; 100 ° C) for 90 minutes extraction time. Frequently stirring during incubation. After incubation, extract the hot acid filtered through nylon / muslin. Then the filtrate is filtered with filter paper. The filtrate was collected then added with twice its volume of absolute ethanol 95%l, then the filtrate precipitate was washed twice with 95% ethanol. The pectin precipitate obtained is dried in an oven at 50°C until it reaches a constant weight. The results of Kepok banana peel extract were calculated as the weight of dry pectin (g) per 100 g of dry weight of banana peel powder. [11,12].

2.3. Experimental by The Central Composite Design of the RSM

The Central Composite Design of the Response Surface Methodology was used to investigate the effects of two independent process variables, namely, pH (A) and temperature (B) and the extraction time set in response, the results of pectin extraction (Y). The range of variables is chosen based on (Table 1), where variables are coded as –α, -1, 0, +1, + α (low, basal, high).

Table 1. Experimental and coded levels of three variables employed for pectin extraction

Variable	**Factor coding**	**Units**	**Levels**				
			-α	-1	0	+1	+α
pH	A	-	1.29	1.50	2.00	2.50	2.71
Temperature	B	°C	51.72	60.00	80.00	100.00	108.28

RSM and CCD are used to standardize extraction parameters. The experimental values obtained for the results of pectin (%) for kepok banana peel in Table 2. Results show in 3D graphs (Figure 1)

shown that with increasing temperature, pH and extraction time (set value; 90 minutes), pectin yield increased. The expected value of the pectin results obtained by using RSM before compared with unused values. This shows that the response of the surface methodology validates experimental data. The Central Composite Design of RSM is used to determine the optimal combination of variables [13].

3. Result and Discussion

The effect of various processes on the results of pectin extraction was investigated with used of a three-dimensional response surface plot.

The polynomial equation generated by the software is as follows: (Eq. 1):

$$\text{Yield (Y)} = X0+X1A+X2B+X3C+X11A2+X22B2+X33C2+X12AB+X13AC+X23BC \quad (1)$$

Table 2. Experimental values with different pH and temperature for CCD pectin results by RSM

Run	Factor A (pH)	Factor B (Temperature) °C	Yield Pectin (%)
1	1.50	60.00	9.79
2	2.71	80.00	14.89
3	2.71	80.00	15.53
4	2.50	100.00	21.21
5	1.50	100.00	7.35
6	2.50	100.00	19.91
7	2.00	80.00	9.75
8	1.29	80.00	11.22
9	1.29	80.00	10.53
10	1.50	100.00	8.22
11	2.00	51.72	7.59
12	2.00	108.28	16.59
13	2.50	60.00	6.84
14	2.00	108.28	15.48
15	2.71	80.00	16.46
16	2.50	60.00	7.55
17	1.50	60.00	10.22
18	2.00	80.00	11.54
19	2.00	51.72	6.38
20	2.00	108.28	16.43
21	2.50	100.00	22.57
22	2.00	80.00	13.37
23	2.00	51.72	5.79
24	1.50	60.00	10.23
25	1.50	100.00	9.22
26	1.29	80.00	9.58
27	2.50	60.00	7.87

Pectin obtained from kepok banana peel was extracted using hot acid and further precipitation by 96% ethanol. The results of pectin depend on the peel and acid used the extraction process, a dry mass of 10 g was subjected to extraction by adding hydrochloric acid with a pH of 1.5 - 2.5 with and

temperatures of 60°C - 100°C. Hydrolysis extracts containing acid from pectin constituents which are insoluble into pectin dissolve at contact with them, resulting in maximum recovery of pectin at acidic pH. Increased pH inhibits pectin release probably due to pectin aggregation. With experimental analysis the probability value (P-value) for each parameter is shown in Table 3.

Table 3. The analysis of variance (ANOVA) for the results of pectin

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	553.16	5	110.63	100.95	< 0.0001	*signifikan
A	116.65	1	116.65	106.44	< 0.0001	
B	244.64	1	244.64	223.22	< 0.0001	
A2	3.43	1	3.43	3.13	0.0916	
B2	0.36	1	0.36	0.33	0.5734	
AB	183.14	1	183.14	167.11	< 0.0001	
Residual	23.02	21	1.10			
Lack of Fit	5.48	3	1.83	1.88	0.1697	
Pure Error	17.53	18	0.97			
Cor Total	576.18	26				

Note:
* p-value <0,005 significant
R^2 = 0.9601;
Adj R^2 = 0.9505
Standard deviation= 1.05

The responses obtained from this experiment, after being analyzed using ANOVA are very similar to the second orderly polynomial model compared to other polynomial models. The regression equation obtained is shown in Equation 1.

The results of the relationship between the ratio of pectin extraction and variants tested based on Response Surface Methodology (RSM) are formulated in the form of regression equations as follows:

Pectin Yield (%) = 11.55 + 2.20*A + 3.19*B + 0.63*A2 - 0.20*B2 + 3.91*A*B (1)

Pectin Yield is the term encoding factor. The Analysis of Variance confirms the model accordingly, when the Prob> F value is less than 0.05 indicating significant. If the P-value is less than 0.001, it shows that it is very significant, which means that this item has a greater influence than the other variables. Regression coefficient values (R^2) 0.9601 and close to 1, indicate that the second-order regression model is fulfilled for the results of the extraction of kepok banana pectin. Each variable factor at the optimal level determined by building a three-dimensional surface plot based on the equation of the mathematical model that has been issued. The value of the extract of kepok banana pectin was reached at 21.57%. This plot also represents the interaction between two variables while maintaining the third variable (Figure 1). The value of 96.01% indicates that the total variation is explained by the model. The results of analysis of variance (ANOVA) are shown in Table 3.

Experimental data from the extraction of kepok banana pectin from analysis of variance showed that the model was quite appropriate and significant for each variable. Optimizing the pH and temperature variables in the process of extracting kepok banana peel pectin statistically showed significant results. The optimized extraction conditions are at pH 2.5 and temperature 100C and the

expected results are 21.28%. This shows that pH and temperature are important factors in maximizing the yield extraction process of kepok banana peel.

The 3-dimensional appearance of the response surface for the extraction of kepok banana pectin is shown in Figure 1, the effect of A (pH) and B (temperature) and the extraction time specified.

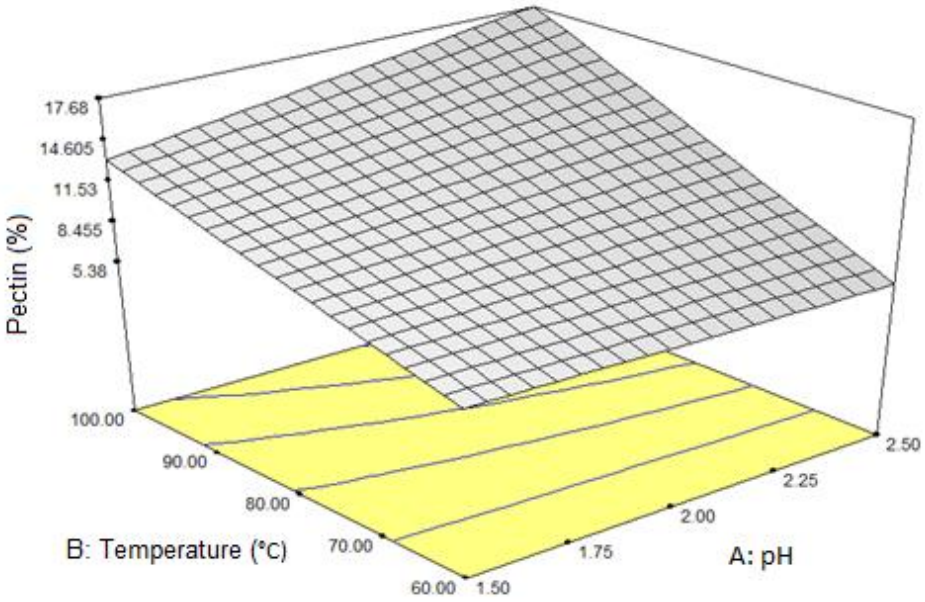

Figure 1. 3-Dimension RSM for kepok banana peel pectin yield as a function of pH and temperature (extraction time 90 minutes).

4. Conclusion

This study emphasizes the extraction of pectin from kepok banana peel. Based on the results and RSM discussion, it can be said that the variable pH and temperature are important and statistically significant factors in the extraction process to maximize the yield of pectin from Kepok banana skin. The best extraction conditions optimized were pH 2.5 and temperature 100oC and the result was 21.57 %%, which was under the predicted extraction conditions of 21.28%. Thus, optimization of pectin extraction technology is feasible.

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